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## Levels and Distribution of Some Persistent organochlorine Pesticide Residues and polychlorinated biphenyls in Water and Soils of Omar AL-Mokhtar Location, Libya.

Salah. M. I. Hassan and Omuklthum.A. A. Abduljalil

Department of Plant Protection (pesticides), Omar Al-Mukhtar University.

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\* Corresponding author E-mail: salah30551@gmail.com

### Abstract

This study reports the concentration levels of the organochlorine pesticide (OCPs) residues in the soil and underground water samples collected from Omar AL-Mokhtar, Libya. A total of 5 soil and 5 underground water samples were collected from the study region. The levels of 7 organochlorine pesticides (OCPs), which included (Heptachlor, Aldrin, Heptachlor Epoxide, Dieldrin, Endosulfan, 4,4-DDT, and Beta endosulfan) and hexachlorocyclohexane (HCH) isomers (alpha, gamma, and delta), were determined, and 7 polychlorinated biphenyls (PCBs) (PCB28, PCB5, PCB101, PCB1188, PCB135, PCB138, PCB180). The OCPs concentrations were determined using gas chromatography (GC) equipped with Electron Capture Detector. The results showed contamination of soil and underground water of the region with several persistent organic pesticides contaminated with the studied pesticides.

**Keywords:** Organochlorine Pesticides, Soil, Underground Water, Polychlorinated Biphenyls, Gas Chromatography.

## **Introduction \**

For the past thirty years, a serious concern has arisen due to the presence of persistent organic pollutants (POPs) in the environment and their threat to the wild life and mankind. As is well known, DDT and PCBs were listed by the Stockholm Convention as 2 of 12 persistent organic pollutants (POPs) in 2004, and more recently,  $\alpha$ -HCH,  $\beta$ -HCH, and  $\gamma$ -HCH (lindane) were added to the list in 2009(12). Persistent organic pollutants (POPs) are a major group of hazardous chemicals having anthropogenic origin and three main characteristics: persistence, bioaccumulation and long range transport(11).  $\alpha$ -HCH, DDT and polychlorinated biphenyls (PCBs) are ubiquitous chemicals and persistent, toxic and bio-accumulative in nature (14). These are long range transport pollutants and can be transported to regions far from their original sources, such as the Organochlorine pesticides (OCPs) (3), are among the most widely applied chemicals in the world, They have been used for pest and insect control in more than half a century(7).

They have a wide range of acute and chronic health effects, including cancer, neurological damage, reproductive disorders, immune suppression, birth defects, and are also

suspected endocrine disruptors (13). Moreover, due to their lipophilic properties and their resistance to biochemical degradation, these compounds tend to accumulate in fatty tissues and biomagnify, reaching harmful concentrations in organisms situated at the high-end of the food chain, such as humans (22). In recent years, there has been a growing interest in these chemicals due to their potential toxicity and adverse impacts on human health (17). Despite the ban on persistent organochlorines (OC) in most of the developed nations since the early 1970s, their uses continued for agricultural and public health purposes in many developing countries owing to their low cost and versatility in industry, agriculture, and public health until very recently (9).

## **Material and methods \**

**Area of study:** Soil and water samples were obtained in April 2019 from location Omar Al-Mokhtar, Libya (Fig 1). Five water samples were collected from groundwater used for irrigation and five soil samples (0-30 cm depth) were collected from the different locations of study using a hand auger.



**Figure (1):** Satellite image of the studied area showing the locations of the collected samples from Omar AL-Mokhtar, Libya.

### **Sample Collection and Preservation:**

**Samples of water:** Water samples were taken at five different locations. The sample was then stored in a pre-cleaned glass bottle. The water samples were acidified by concentrated HNO<sub>3</sub> to pH 2 to prevent alteration of the organic matter. The samples were kept in a glass bottle and preserved in a refrigerator prior to analysis.

**Samples of soil:** Were taken at five different locations. The sample was taken into a glass bottle, labeled as represented. The sediment samples were air-dried, sieved through 2mm mesh, and stored in black polyethylene bags prior to subsequent analysis.

### **Chemical Analysis:**

#### **Extraction of OCPs from Water Sample:**

Method 3510 as described by US EPA 2007(21) was used to extract the pesticide residues in the water samples. 50 ml of dichloromethane was introduced into the separating funnel containing 100 ml of the

sample and shaken vigorously for about 2 minutes. The sample was allowed to settle for 30 minutes to ensure the separation of the phases. After separation, the organic layer was filtered into a 250 ml conical flask through anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) that has been prewashed with dichloromethane. The extractions were repeated twice using a 50 ml portion of dichloromethane and later combined. The combined organic extracts were concentrated using a rotary evaporator at 45°C and low pressure. 5 ml of n-hexane was added to the extract in dichloromethane to exchange the solvent. The extracts were further concentrated to 1-2 ml using a rotary evaporator at 45°C until no further dichloromethane remained in the extract. The extracts were transferred into 2 ml GC vials before analysis with Gas Chromatography.

#### **Extraction of OCPs from Soil Sample:**

Extraction of OCPs in Soil samples was carried

out by the EPA 3550C method described by USEPA (2000) (20). For the Soil, 20 g of each sample and 20g of anhydrous Na<sub>2</sub>SO<sub>4</sub> were added. A 50 ml mixture of acetone and n-hexane (1:1 v/v) were then mixed with the sample in a 100 ml volumetric flask. This was followed by sonication in a high-frequency ultra-sonic bath for 10-15 minutes at about 60°C. The extract was then decanted into a round bottom flask. The extraction process was repeated with additional 50ml (acetone and n-hexane), sonicated and allowed to settle and decant into the same round bottom flask. The extracts were concentrated using a rotary evaporator to 1-2 ml. The extract was re-dissolved in 5 ml n-hexane and later concentrated to 2 ml in a rotary evaporator at 40°C.

**Extract Clean-up:** Soil samples were cleaned-up using activated silica gel. The column of about 15 cm (length) X 1cm (internal diameter) was packed with 2 g of deactivated silica gel and 1g anhydrous Na<sub>2</sub>SO<sub>4</sub> packed on top of the silica gel (adsorbent). The columns were each conditioned with 15 ml n-hexane before clean-up. The extract was introduced into the column and eluted with 20 ml of n-hexane and diethyl ether (1:1 v/v). The eluate was concentrated to dryness on the rotary evaporator and recovered into 2 ml n-hexane. The extracts were transferred into glass GC vials for GC analysis using an electron capture detector (ECD) (4).

**Identification of OCPs and PCBs:** All samples and blank were analyzed by Gas

Chromatograph-Mass Spectrometer, GCMS (Thermo Scientific Company -Trace DSQ II MS) on fused silica capillary column (Thermo TR-35 MS [3.0m\* 0.25mm] filled with 35% phenyl polyphenylene-siloxane (stationary phase). Organochlorine insecticides were quantified from individually resolved peak areas with the corresponding peak areas of the standards. Helium was used as carrier gas at a flow rate of 2 ml/min. The temperature was programmed from 90–140° C with a rate of 5° C/min, then hold at 140 °C for 1min, and from 140-250° C with a rate of 3° C/min and was hold at 250 °C for 1min, and from 250-300° C with a rate of 20° C/min and was hold at 300° C for 1min. The injector, ion source, and detector temperatures were set at 280° C, 250° C, and 310° C, respectively. Three microliters (3µl) of each sample was injected in the split-split less mode and the purge time was 1 min (10).

**Statistical analysis:** Statistical analyses were carried out by analysis of variance (ANOVA) using Minitab version 18 software. Statistical differences in pesticide were analyzed by t-test between soil and underground water samples. Also, differences in pesticide were analyzed by one way between underground water samples in different pesticides, also between in soil samples. Differences were considered significant at p values ≤ 0.05 after which, the Tukey's HSD (honestly significant difference) analysis was performed to determine whether

there were statistically significant differences p values  $\leq 0.05$  between different pesticides and different samples.

## **Results and discussion \**

The results of the investigation have indicated the presence of certain organochlorine pesticides in surface soil and underground water samples collected at the investigation points, such as (Heptachlor, Aldrin, Heptachlor Epoxide, Dieldrin, Endosulfan, 4,4-DDT, and Beta endosulfan) and hexachlorocyclohexane (HCH) isomers (alpha, gamma, and delta), were determined and 7 polychlorinated biphenyls (PCBs) (PCB28, PCB5, PCB101, PCB1188, PCB135, PCB138, PCB180). surface soil samples were found to be more contaminated than groundwater concentrated pesticide residues.

**Results underground water and soil samples:** The average concentrations of organochlorine and PCB pesticides in water and soil samples were collected from Omar Mokhtar location, Libya. Analyzed to assess the levels of organochlorine and PCB pesticides contamination based on these samples collected from five different locations (soil and groundwater) during December month. The results of water and soil samples analysis from Omar Mokhtar's location during winter (2019) affirmed the presence of organochlorine and PCB pesticides in different locations. Water samples collected from five

locations from groundwater and soil samples of five different locations show different contamination levels of concentrations of organochlorine and PCB residues in each location under study.

Water samples collected from 7 groundwater supplies of different locations show different contamination levels of organochlorine and PCB. Monitored pesticides in this study were detected with values less than MRL's according to World Health Organization (WHO) and the Libyan National Center for Standardization and Metrology (LNCSM) (23) in all samples collected.

The results showed that table (1) the values of organochlorine pesticide residues in water samples, where was Heptachlor residue ranged from location 1 (0.0040ng/l) to location 5 (0.0045 ng/l). In Aldrin residue ranged location 1(0.0053 ng/l) to location 5 (0.0042 ng/l). Also, in Heptachlor Epoxide residue values ranged samples ranged from location 1 (0.0047ng/l) to location 5 (0.0039ng/l). The results showed that the Dieldrin residues values ranged from location 1 (0.0052 ng/l) to location 5 (0.0031 ng/l) and in Endosulfane ranged from location 1 (0.0052 ng/l ) to location 5 (0.0029 ng/ml),in addition water samples content 4,4-DDT residue where ranged location 1 (0.0013 ng/l ) to location 5 (0.0016 ng/ml) excluding location 4 was not non detected from any pesticides. In Beta endosulfan residue ranged from location 1 (0.0026 ng/l) to location 5 (0.0031 ng/l) and Alpha – HCH residue ranged from location 1

(0.011 ng/l) to location 5 (0.0027 ng/l) including location 4 was not non detected from any pesticides. Also, gama - HCH residue ranged from location 1 (0.0032 ng/g) to

location 5 (0.0033 ng/l). Finally, Delta - HCH ranged from location 1 (0.0021 ng/l) to location 5 (0.0034 ng/l) in all water samples under study.

**Table 1.** Concentration (ng/l) of organochlorine pesticide residues in water samples.

Pesticides	location 1	Location 2	Location 3	Location 4	Location 5	WHO/ (LNCSM)
Heptachlor	0.0040	0.0034	0.0036	0.0027	0.0045	0.00003
Aldrin	0.0053	0.0036	0.0040	0.0034	0.0042	0.00003
Heptachlor Epoxide	0.0047	0.0039	0.0031	0.0021	0.0039	0.00003
Dieldrin	0.0036	0.0021	0.0025	0.0025	0.0031	0.00003
Endosulfan	0.0052	0.0038	0.0033	0.0016	0.0029	0.020
4,4-DDT	0.0013	0.0011	0.0009	ND	0.0016	0.00001
Beta endosulfan	0.0026	0.0024	0.0026	0.00105	0.0020	0.020
Alpha - HCH	0.0011	0.0009	0.0010	ND	0.0027	0.002
gama - HCH	0.0032	0.0021	0.0025	0.0017	0.0032	0.002
Delta - HCH	0.0021	0.0017	0.0020	0.0020	0.0034	2

ND: Not detected

The results showed that tables (2) the values of Concentration (ng/g) PCB residues in water samples. For PCB28 residue ranged from location 1 (0.0014 ng/g) to location 5 (0.0015 ng/g) excluding location 4 was not non detected from any pesticides. In PCB52 residue ranged location 1 (0.0019 ng/g) to location 5 (0.0014 ng/g). Also, in PCB101 residue values ranged samples ranged from location 1 (0.0010ng/g) to location 5 (0.0015

ng/g). The results showed that the PCB118 resides values ranged from location 1 (0.0007 ng/g) to location 5 (0.0013 ng/g) and in PCB135 ranged from location 1 (0.0012 ng/g) to location 5 (0.0012 ng/g), in addition water samples content PCB138 residue where ranged location 1 (0.0014 ng/g) to location 5 (0.0014 ng/g). Finally, in PCB180 reside ranged from location 1 (0.0009 ng/g) to location 5 (0.0013 ng/g) in samples under study.

**Table 2.** Concentration (ng/l) PCB residues in water samples.

Pesticides	location 1	Location 2	Location 3	Location 4	Location 5	WHO/LNCSM
PCB28	0.0014	0.0010	0.0012	ND	0.0015	0.5
PCB52	0.0019	0.0012	0.0015	0.0007	0.0014	
PCB101	0.0010	0.0014	0.0014	0.0008	0.0015	
PCB118	0.0007	0.0005	0.0014	0.0009	0.0013	
PCB135	0.0012	0.0010	0.0013	0.0005	0.0012	
PCB138	0.0014	0.0015	0.0012	0.0005	0.0014	
PCB180	0.0009	0.0005	0.0015	0.0006	0.0013	

ND: Not detected

Tables (3) shown the result of the analysis of organochlorine pesticide residues in soil samples collected from five locations during December .Where was Heptachlor residue ranged from location 1 (0.095 ng/g) to location 5 (0.041ng/g). In Aldrin residue ranged location 1(0.053 ng/g) to location 5 (0.0083 ng/g). Also, in Heptachlor Epoxide residue values ranged samples ranged from location 1 (0.0072ng/g) to location 5 (0.0099ng/g). The results showed that the Dieldrin resides values ranged from location 1 (0.0052 ng/g) to location 5 (0.005 ng/g) and in Endosulfane ranged from location 1 (0.011

ng/g) to location 5 (0.015 ng/g), in addition water samples content 4,4-DDT residue where ranged location 1 (0.005 ng/g) to location 5 (0.001 ng/g). In Beta endosulfan reside ranged from location 1 (0.018 ng/g) to location 5 (0.021 ng/g) and Alpha – HCH residue ranged from location 1 (0.062 ng/g) to location 5 (0.005 ng/g). Also, gama - HCH residue ranged from location 1 (0.071 ng/g) to location 5 (0.0433ng/g). Finally, Delta – HCH ranged from location 1 (0.087 ng/g) to location 5 (0.011 ng/g) in all water samples under study.

**Table 3.** Concentration (ng/g) of organochlorine pesticide residues in soil samples.

Pesticides	location 1	Location 2	Location 3	Location 4	Location 5
Heptachlor	0.095	0.055	0.027	0.032	<b>0.041</b>
Aldrin	0.053	0.053	0.005	0.085	<b>0.083</b>
Heptachlor Epoxide	0.072	0.031	0.075	0.063	<b>0.099</b>
Dieldrin	0.005	0.087	0.003	0.098	<b>0.005</b>
Endosulfan	0.011	0.015	0.057	0.081	<b>0.015</b>
4,4-DDT	0.005	0.061	0.059	0.024	<b>0.002</b>
Beta endosulfan	0.018	0.041	0.066	0.060	<b>0.021</b>
Alpha – HCH	0.062	0.016	0.008	0.027	<b>0.005</b>
gama - HCH	0.071	0.001	0.095	0.087	<b>0.043</b>
Delta – HCH	0.087	0.075	0.061	0.023	<b>0.011</b>

**ND: Not detected**

Tables (4) shown the concentrations of PCB residues in soil samples during December collected from different locations during December. The values of PCB28 residue ranged from location 1 (0.526 ng/g) to location 5 (0.2115 ng/g). In PCB52 residue ranged location 1 (0.3565 ng/g) to location 5 (0.2985 ng/g). Also, in PCB101 residue values ranged samples ranged from location 1 (0.3435 ng/g) to location 5 (0.2655 ng/g). The

results showed that the PCB118 resides values ranged from location 1 (0.4055ng/g) to location 5 (0.394ng/g) and in PCB135 ranged from location 1 (0.2315 ng/g) to location 5 (0.324 ng/g), in addition soil samples content PCB138 residue where ranged location 1 (0.4355ng/g) to location 5 (0.4355ng/g). Finally, in PCB180 reside ranged from location 1 (0.3165ng/g) to location 5 (0.2605ng/g) in samples under study.

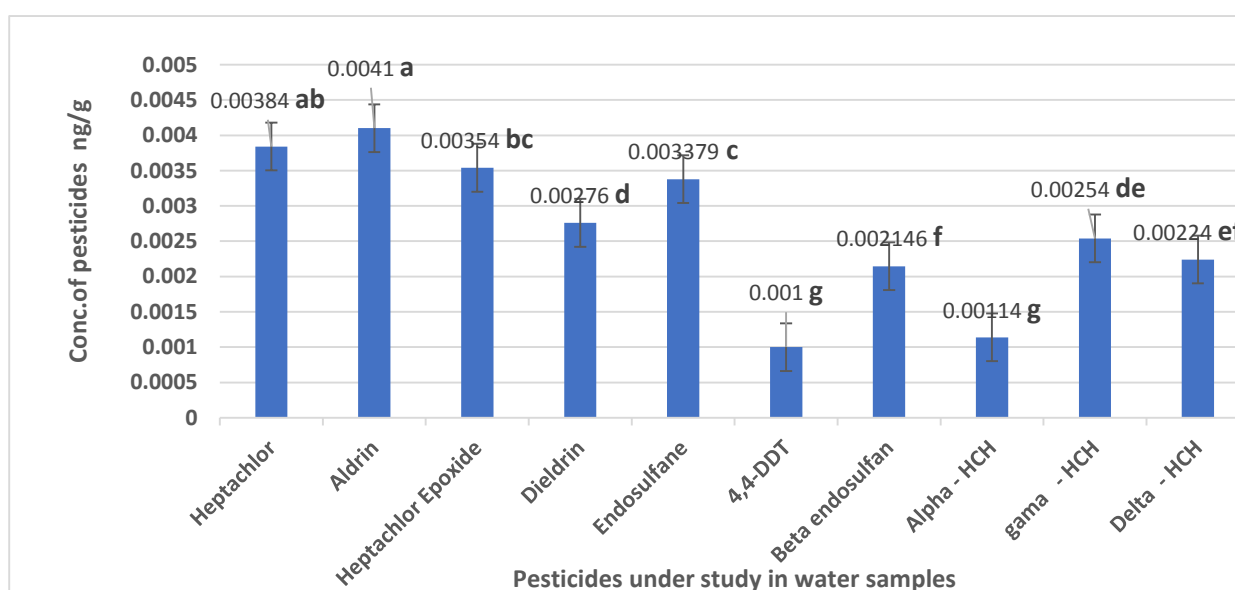
**Table 4.** Concentration (ng/g) of PCB residues in soil samples.

Pesticides	location 1	Location 2	Location 3	Location 4	Location 5
PCB28	0.526	0.4275	0.4275	0.3595	<b>0.2115</b>
PCB52	0.3565	0.3945	0.3945	0.2845	<b>0.2985</b>
PCB101	0.3435	0.253	0.253	0.3375	<b>0.2655</b>
PCB118	0.4055	0.339	0.339	0.283	<b>0.394</b>
PCB135	0.2315	0.3555	0.3555	0.445	<b>0.324</b>
PCB138	0.4355	0.256	0.256	0.3585	<b>0.4355</b>
PCB180	0.3165	0.346	0.346	0.215	<b>0.2605</b>

ND: Not detected

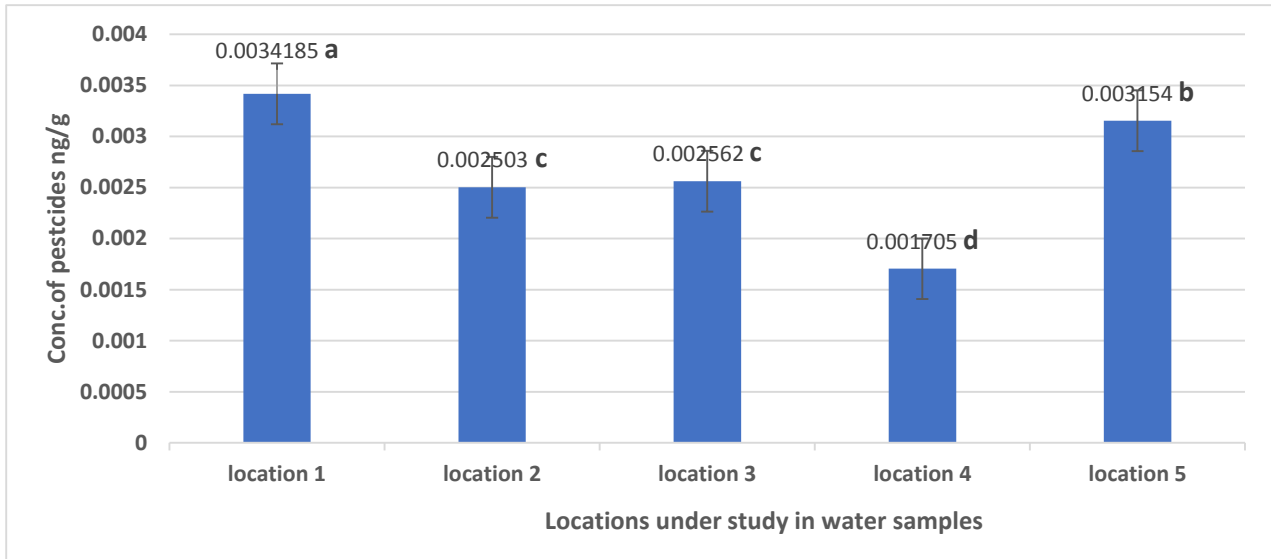
In general, figure (2) shown organochlorine pesticide residues in water samples residue in different locations by using analysis of variance and there were significant differences between Aldrin and other pesticides at ( $p < 0.05$ ).

Analysis of variance (ANOVA) for groundwater, which is presented in figure (3) showed a significant difference between locations at ( $p < 0.05$ ).



**Figure (2).** Shows the mean concentration of organochlorine pesticide residues in water samples.

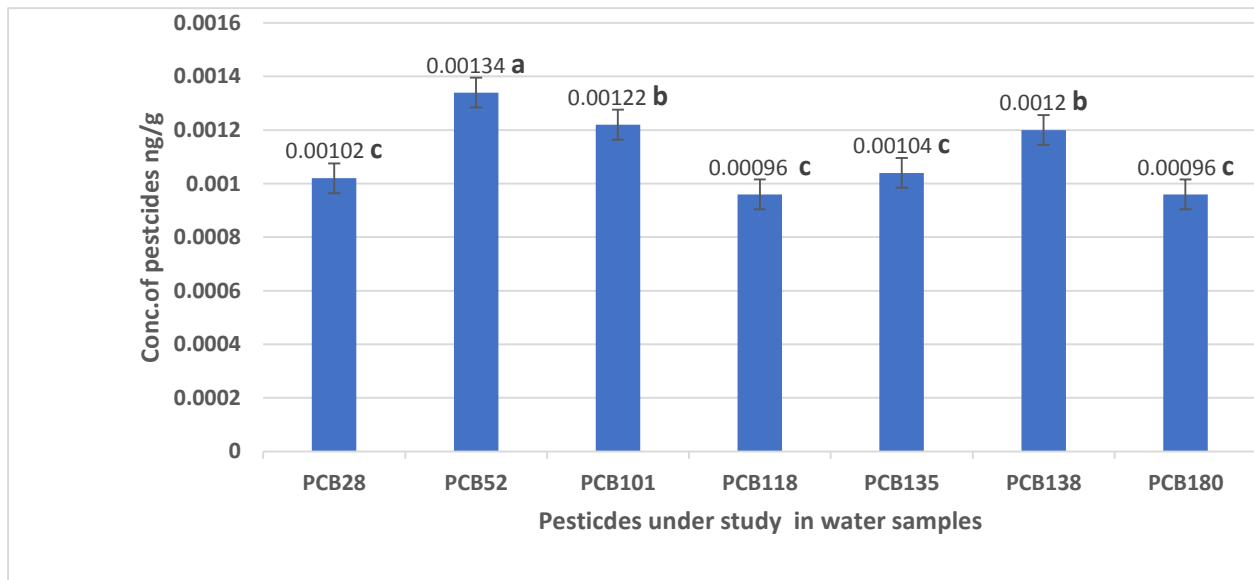




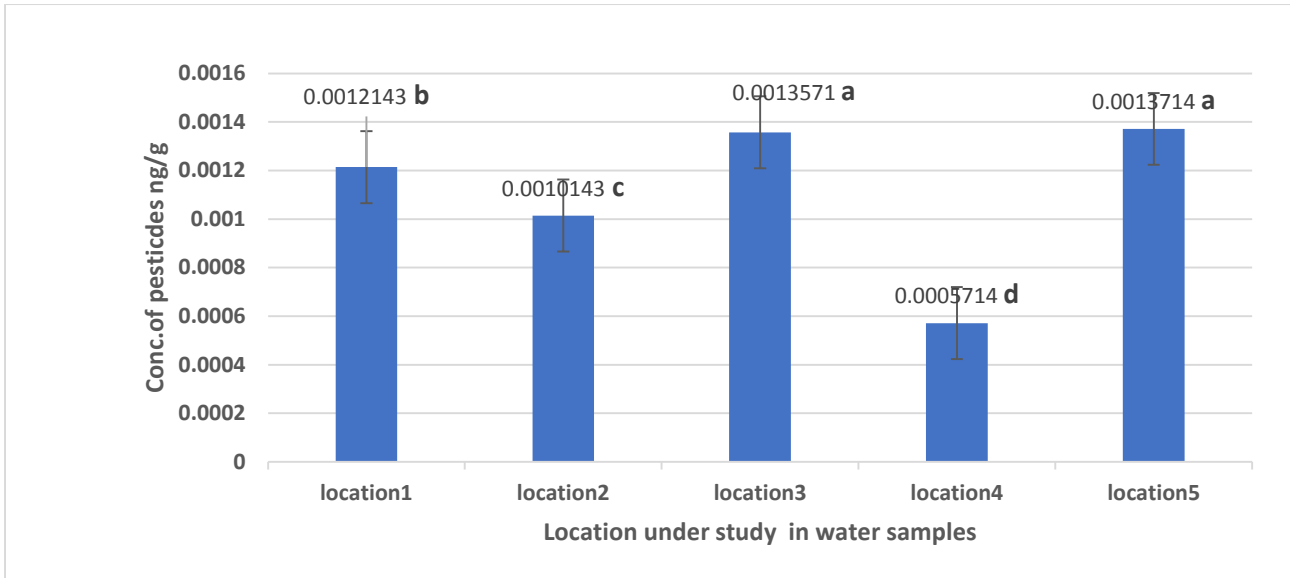
**Figure (3).** Shows the mean concentration of organochlorine pesticide residues in different locations.

Figures (4) shown the concentrations of PCB residues in water samples by using analysis of variance and there were significant differences

between PCB52 and other pesticides the which is presented in figures (5) showed a significant difference between locations at ( $p < 0.05$ ).



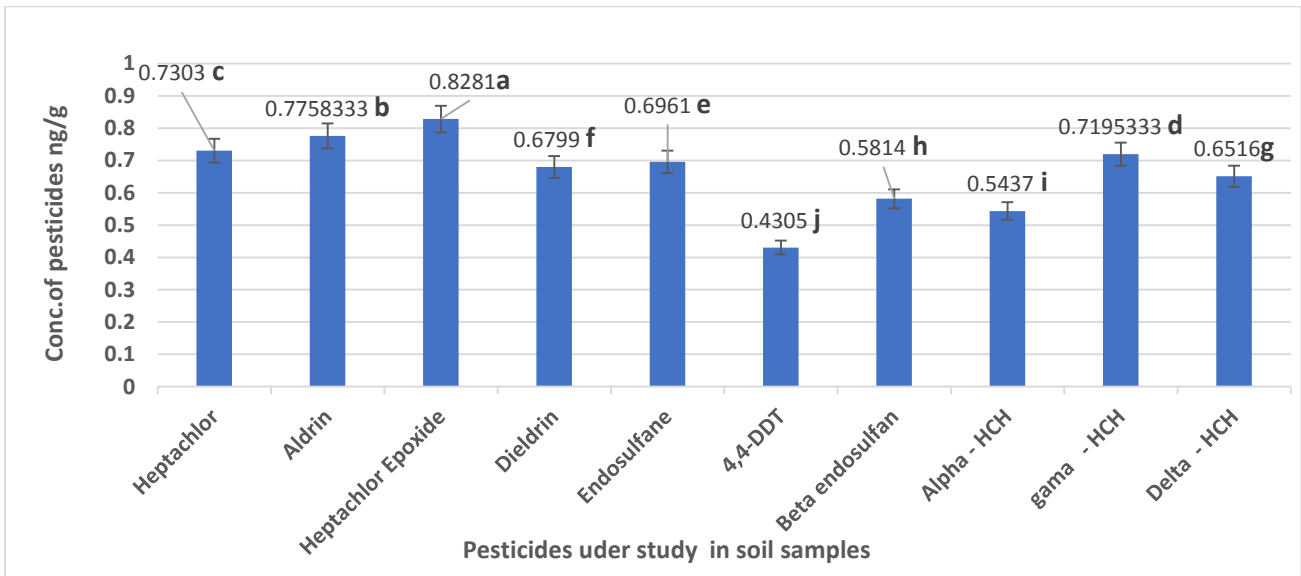
**Figure (4).** Shows the mean concentrations of PCB pesticide residues in water samples.



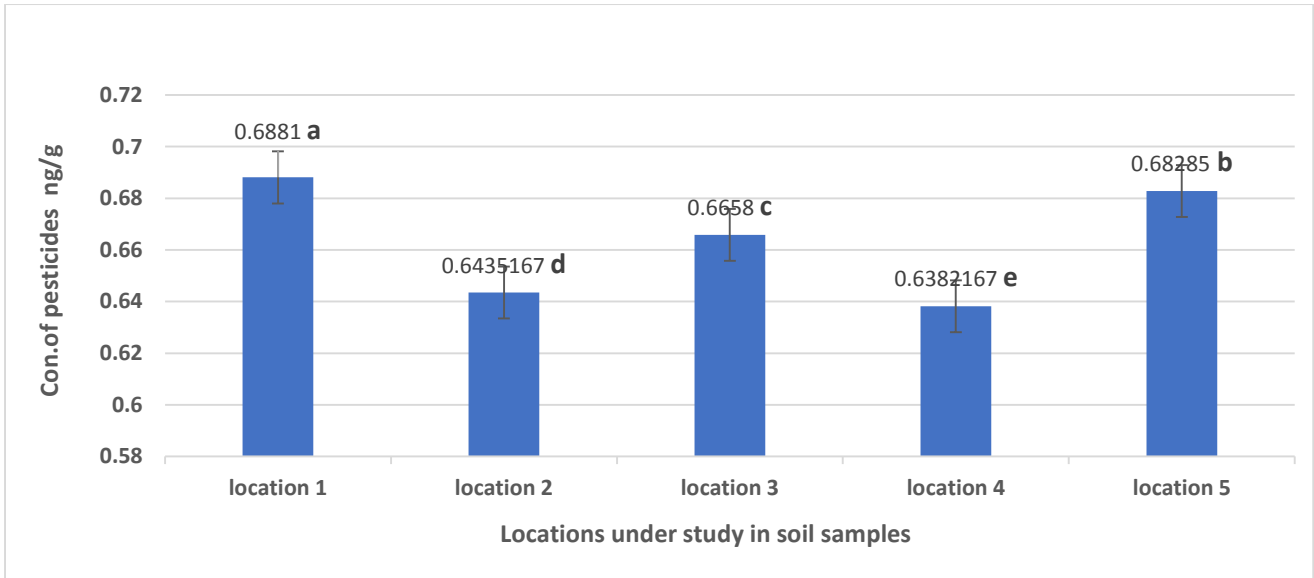
**Figure (5).** Shows the mean of concentrations of PCB residues in different locations.

The concentration of organochlorine pesticide residues in soil samples were different between Heptachlor Epoxide than other pesticides in different locations as shown in figures (6),

showed a significant difference at ( $p < 0.05$ ), On the other hand, the concentration of organochlorine pesticide residues between locations at ( $p < 0.05$ ), as shown in figures (7).



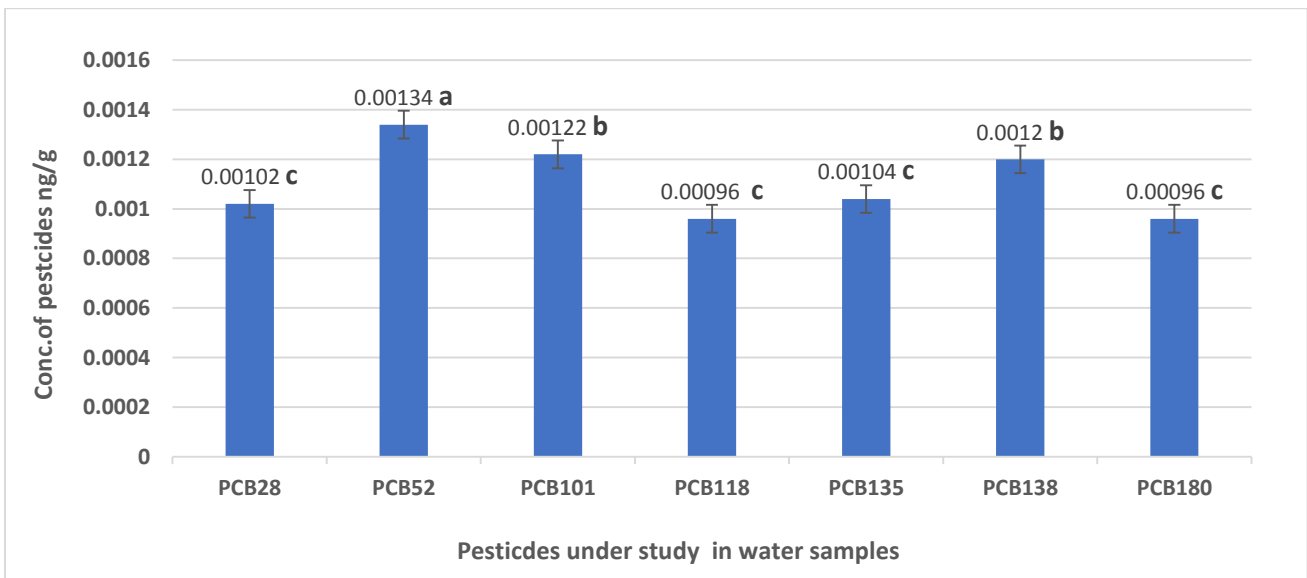
**Figure (6).** Shows the mean concentration of organochlorine pesticide residues in soil samples.



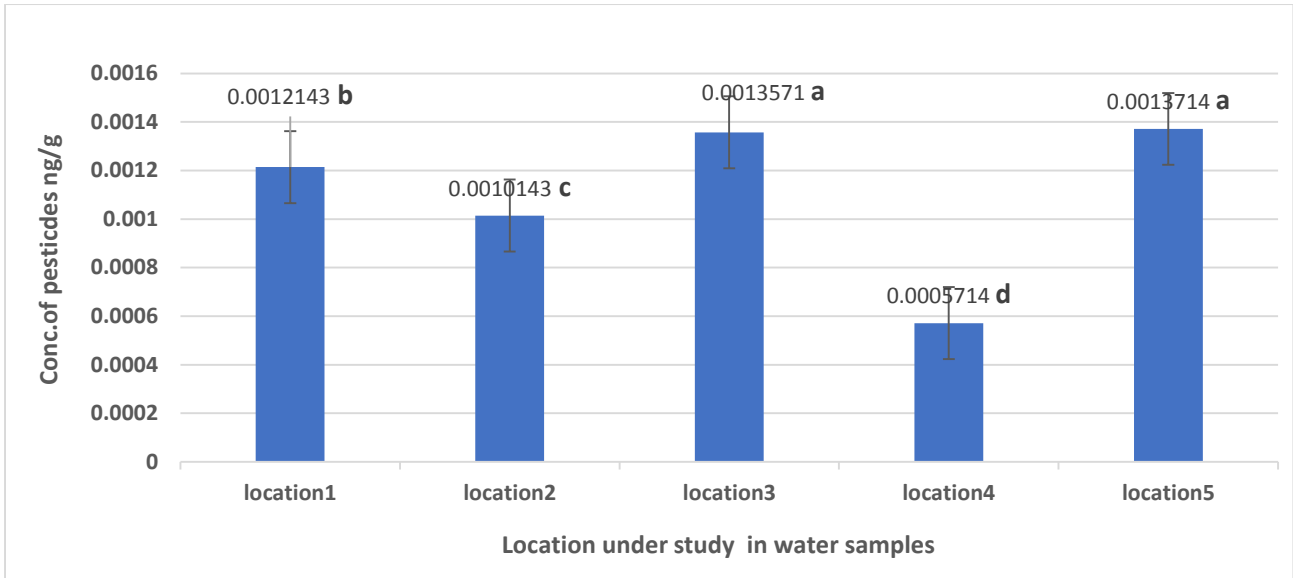
**Figure (7).** Shows the mean concentration of organochlorine pesticide residues in different locations.

Figures (8) shown the concentration of PCB residues in soil samples by using analysis of variance and there were significant differences between PCB52 and different locations at ( $p <$

0.05), which is presented in figures (9) showed a significant difference between regions at ( $p < 0.05$ ).



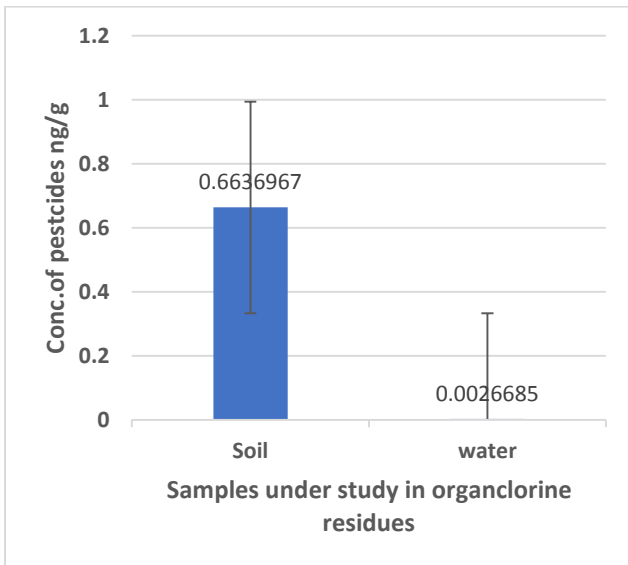
**Figure (8).** Shows the mean concentrations of PCB pesticide residues in soil samples.



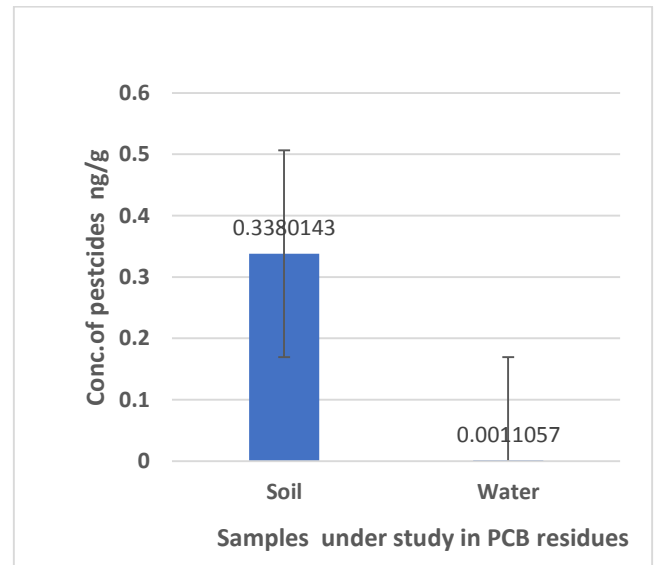
**Figure (9).** Shows the mean of concentrations of PCB residues in different locations.

In the t-test in the mean levels concentration of organochlorine pesticide residues was observed that a significant difference between soil and groundwater at a concentration of each pesticide between locations at ( $p < 0.05$ ) as

shown in figure (10). There were differences between soil and groundwater in PCB residues as shown in figure (11). Showed a significant difference at ( $p < 0.05$ ).



**Figure (10).** Analysis of variance between soil and groundwater samples.



**Figure (11).** Analysis of variance between soil and groundwater samples.

Generally, low levels of residues were found in areas associated with agricultural pesticide use but the levels in the former storage areas were substantially high. organochlorine and PCB pesticides were dominant in all the studied locations (21).

This study is in agreement with (18) findings which showed. The probable reason for higher residues in December may have been due to most of these organochlorines had virtually phased out many years ago and their presence in water residues from the past application and this is due to firstly, the persistent nature of these compounds.

Based on our spatiotemporal monitoring, the detected pesticides in water samples collected from different water resources exhibited fluctuations due to residential and agricultural activities along with sampling points. Also, maybe this contamination is caused by the increasing amount of organochlorine application and also an improper way of disposal wastewater into the environment. The resulting agreement with (5;15).

In addition, biodegradation occurs under both aerobic and anaerobic conditions and is the major degradation process for PCBs in soil. No abiotic process is known to significantly degrade PCBs in soil; however, photolysis of PCBs on surface soil may occur. (8) Who reported levels PCB in similar studies were lower than those obtained in this study. Provide a review of biodegradation of PCBs in soil. Experiments with pure and mixed cultures of

microorganisms show that some congeners of PCBs, usually containing six or fewer chlorine substituents, biodegrade under aerobic conditions (6). Biodegradation rates are highly variable because they depend on several factors, including the amount and location of chlorination, PCB concentration, type of microbial population, available nutrients, and temperature. The most common process for the aerobic degradation of PCBs by bacterial cultures proceeds by two distinct steps, one involving bioconversion of PCBs to chlorinated benzoic acids and the other involving mineralization of chlorobenzoates to carbon dioxide and inorganic chlorides This finding is consistent with (16). Complete mineralization of biodegradable PCBs requires the presence of two clusters of genes for the two-step bioconversion process (19), therefore, complete degradation requires mixed microbial cultures (2).

In the same vein, the detection frequency and concentration level of organochlorine pesticides were higher in soil than in water, also PCB pesticides were higher in the soil in locations under study this is due to the high persistence of organ chlorine pesticides (OC), due to itself most of the applied pesticides undergo too many transformation processes. This study supports evidence from previous observations (15).

Analysis of results in water samples in this study showed that levels of most organochlorine detected were the minimum,

from levels for water, organochlorine, and PCB residues. From these results we can conclude that organochlorine and PCB residues were more significant in December this may be in the first stage drift outside of the intended area when it is sprayed during December, this may percolate, or leach, through the soil, it may be carried to the water as runoff as in the case of December, or it may be spilled, for example accidentally or through negligence by (24).

### Conclusions \

The results of groundwater and soil samples analyzed from different locations at the Omar Mokhtar, Libya during December month affirmed the presence of organochlorine and PCB pesticides in different locations in all water and soil samples. It is noteworthy that location 4 was not detected in 4,4-DDT and Alpha – HCH of groundwater. This study has not shown alarming levels of organochlorine and PCB pesticides in different locations where the contamination registered in December, where observed pesticides in this study were detected with values under limit than MRL's according to World Health Organization (WHO) and the Libyan National Center for Standardization and Metrology (LNCSM). Toxicological and water quality standards data revealed the hazardous risk of detected pesticides in the Libya groundwater to human and aquatic life. Thus, our monitoring data will provide viewpoints by which stricter legislation

and regulatory controls can be admitted to avoid groundwater pesticide water pollution.

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## مستويات وتوزيع بعض بقايا مبيدات الآفات الكلورية العضوية الثابتة وثنائي الفينيل متعدد الكلور في المياه والتربة في موقع عمر المختار ، ليبيا.

صلاح محمد ادريس و أم كلثوم أحمد عبد الجليل  
قسم وقاية النبات, كلية الزراعة, جامعه عمر المختار

### الملخص/

توضح هذه الدراسة مستويات بقايا المبيدات العضوية الكلورية (OCPs) في عينات التربة والمياه الجوفية التي تم جمعها من منطقة عمر المختار ، ليبيا. تم جمع ما مجموعه 5 عينات من التربة و 5 عينات من المياه الجوفية من منطقة الدراسة. كانت مستويات 7 من مبيدات الآفات العضوية الكلورية (OCPs) ، والتي تضمنت Heptachlor, Aldrin) Heptachlor Epoxide, Dieldrin ، والتي تضمنت (Alpha HCH و Endosulfan,4,4-DDT Beta endosulfan, Alpha - gama HCH ,Delta HCH و 7 مركبات ثنائي الفينيل متعدد الكلور (PCB180 و PCB138 ، PCB135 ، PCB1188 ، PCB101 ، PCB5 ، PCBs PCB28). تم تحديد تركيزات OCPs باستخدام كروماتوجرافيا الغاز (GC) المجهزة بكاشف التقاط الإلكترون. أظهرت النتائج تلوث التربة والمياه الجوفية للمنطقة بالعديد من المبيدات العضوية الثابتة الملوثة بالمبيدات المدروسة للعينات المدروسة.

**الكلمات المفتاحية :** المبيدات العضوية الكلورية ، التربة ، المياه الجوفية ، مركبات ثنائي الفينيل متعدد الكلور ، الكروماتوجرافيا الغازية.